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(54) **METHOD FOR ANALYZING CONTENT AND DISTRIBUTION OF BORON INTRODUCED INTO POSITIVE ELECTRODE ACTIVE MATERIAL**

VERFAHREN ZUR ANALYSE DES GEHALTS UND DER VERTEILUNG VON BOR, DAS IN EIN POSITIVELEKTRODENAKTIVMATERIAL EINGEFÜHRT WURDE

PROCÉDÉ D'ANALYSE DE TENEUR ET DE DISTRIBUTION DE BORE INTRODUIT DANS UN MATÉRIAU ACTIF D'ÉLECTRODE POSITIVE

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Description**Technical Field**

5 **[0001]** The present invention relates to an analysis method of distribution pattern and content for each distribution position of boron introduced into a cathode active material for a lithium secondary battery.

Background Art

10 **[0002]** Lithium secondary batteries generally have a structure including an electrode assembly composed of a cathode and an anode comprising electrode active materials capable of intercalating/releasing lithium ions and a separator interposed between the two electrodes, and an electrolyte solution which is a transfer medium for lithium ions. Lithium ions move between the anode and the cathode through the electrolyte, causing charging and discharging of batteries.

15 **[0003]** The secondary batteries undergo problems such as deterioration of life characteristics, decomposition of electrolytes or deterioration of active materials during repeated charging and discharging procedures. In addition, when impurities are present on the surfaces of the active materials during the manufacture of electrodes of secondary batteries, the batteries are subject to swelling due to the reaction between impurities and the electrolyte and change over time.

20 **[0004]** In order to overcome the above problems, the electrode active material may be coated or doped with various materials. For example, when lithium transition metal oxides such as lithium-nickel-manganese-cobalt-based oxides are used as the cathode active material, coating or doping with boron results in formation of oxygen-sealing structures, thereby capacity reduction can be slowed down. In addition, boron contained in the cathode active material may enhance structural stability by strengthening the bonding between transition metals and oxygen. In general, the introduction of boron into the cathode active material is carried out by mixing the cathode active material and boric acid (H_3BO_3) and firing at a high temperature wherein boron may penetrate to the inside of the cathode active material or may remain in the form of Li-B-O compound on the surface and outside thereof.

25 **[0005]** In order to evaluate effect of boron on performance of a cathode active material, it is important to understand distribution pattern of boron inside and outside the active material, and in particular, to measure content of boron penetrating to the inside. The current method for analyzing components of a cathode active material is to perform measurement by inductively coupled plasma optical emission spectroscopy (ICP-OES) after pretreatment with acid. In this method, it is possible to quantify even the boron that has penetrated to the inside of primary grains of the structurally stable cathode active material by using acid.

30 **[0006]** However, a technique for separately analyzing boron inside and outside of the cathode active material has not yet been developed.

35 **[0007]** WO 2020/230424 A1 describes a positive electrode active substance for a non-aqueous electrolyte secondary battery that contains a lithium-containing transition metal oxide having secondary particles formed by aggregation of primary particles and at least a tungsten compound and a boron compound that are present between primary particles.

40 **[0008]** US 2018/131006 A1 describes positive electrode active material of secondary particles formed by aggregation of primary particles of a lithium transition metal oxide containing Ni and Mn and that include a boron compound present in the inner part and surface of the secondary particles.

45 **[0009]** US 2016/013476 A1 describes a method of preparing a cathode active material including coating a surface of a lithium transition metal oxide with a lithium boron oxide by dry mixing the lithium transition metal oxide and a boron-containing compound and performing a heat treatment.

50 **[0010]** US 2019/312279 A1 describes a positive electrode active material for a nonaqueous electrolyte secondary battery that contains a first lithium-metal composite oxide containing a secondary particle formed of a plurality of flocculated primary particles and either one or both of a first compound containing lithium and boron and a second compound containing lithium and tungsten.

Detailed Description of the Invention**Technical Problem**

55 **[0011]** Accordingly, the present invention provides an analysis method of content for each distribution position of boron introduced to improve performance of a cathode active material used in a lithium secondary battery.

Solution to Problem

60 **[0012]** According to the invention, there is provided an analysis method of content of boron introduced into a cathode active material, the method comprising:

(S1) preparing a cathode active material sample into which boron is introduced;
 (S2) dissolving the cathode active material sample in water to obtain a first liquid layer and a first precipitate and treating the first liquid layer with acid, and then analyzing the resulting solution by inductively coupled plasma optical emission spectroscopy (ICP-OES) to determine the concentration of boron;
 (S3) dissolving the first precipitate in water to obtain a second liquid layer and a second precipitate and treating the second liquid layer with acid, and then analyzing the resulting solution by ICP-OES to determine the concentration of boron; and
 (S4) adding acid and hydrogen peroxide to the second precipitate, and then analyzing the resulting solution by ICP-OES to determine the concentration of boron.

Effect of the Invention

[0013] According to the present invention, it is possible to sequentially extract and analyze boron introduced to improve performance of a cathode active material for a lithium secondary battery by using a difference in solubility in water and an acid according to the distribution position of boron, thereby determining the optimal content of boron required for improving performance of the cathode active material through the distribution pattern of boron in the cathode active material.

Brief Description of Drawings

[0014]

Fig. 1 illustrates a distribution pattern of boron introduced to the cathode active material.

Fig. 2 shows the extraction results of boron and lithium according to the dissolution time in water of the cathode active material sample in the analysis process according to the embodiment.

Best Mode for Carrying out the Invention

[0015] Hereinafter, the terms or words used in the present specification and claims should not be construed as being limited to conventional or dictionary meanings, and should be interpreted as meanings and concepts consistent with the technical spirit of the present invention based on principles that the inventor can appropriately define the concept of a term to best describe his invention.

[0016] In addition, the configuration shown in the embodiments and drawings described in this specification is only the most preferred embodiment of the present invention and does not represent all of the technical idea of the present invention, which is defined in claim 1.

[0017] The present invention relates to an analysis method of content of boron introduced to improve the performance of a cathode active material for a lithium secondary battery according to a distribution position. Hereinafter, the method will be described in detail for each step.

[0018] First, a cathode active material sample into which boron is introduced is prepared (S1).

[0019] Specifically, the cathode active material sample may be prepared by dry mixing a boron-containing compound and a cathode active material, followed by firing at a high temperature.

[0020] The boron-containing compound may comprise at least one compound selected from the group consisting of H_3BO_3 , B_2O_3 , $C_6H_5B(OH)_2$, $(C_6H_5O)_3B$, $[CH_3(CH_2)_3O]_3B$, $C_{13}H_{19}BO_3$, $C_3H_9B_3O_6$, and $(C_3H_7O)_3B$.

[0021] The cathode active material may comprise a lithium transition metal oxide represented by the following formula 1.



[0022] In the formula, M is one or two or more selected from the group consisting of Al, Zr, Zn, Ti, Mg, Ga and In; $0 \leq x \leq 1.0$, $0 \leq y \leq 0.6$, $0 \leq z \leq 0.6$ and $0 \leq v \leq 0.1$.

[0023] In addition, the firing may be carried out near the melting point of the boron-containing compound, for example at 130 °C to 300 °C, such as 130 °C to 200 °C, for 3 hours to 10 hours. When the firing temperature satisfies the above temperature range, the boron-containing compound is sufficiently melted and non-uniform reaction due to excessive temperature can be prevented.

[0024] After the firing process, boron may be coated on the surface of the cathode active material or penetrate to the inside of the cathode active material to be distributed between primary grains or secondary grains. Referring to Fig. 1, boron is introduced into the cathode active material such that it exists in the coating layer on the surface (A), at grain boundary (B), or in lattice (C) to form lithium boron oxides such as $LiBO_2$, LiB_4O_7 , etc.

[0025] The solubility of boron in water may vary depending on the distribution position of the boron in the cathode

active material. For example, boron present in the coating layer (A) of the cathode active material is readily dissolved in water, while boron present at grain boundary (B) is dissolved in water over a long period of time and boron doped in lattice (C) is hardly dissolved in water and can be dissolved through acid treatment. Therefore, in the present invention, boron present in the cathode active material sample is sequentially extracted by using the difference in solubility for each distribution position.

[0026] In particular, the cathode active material sample is dissolved in water to obtain a first liquid layer and a first insoluble precipitate, and then the first liquid layer is treated with acid (S2). At this time, dissolving the sample in water may be performed at room temperature for 1 to 10 minutes.

[0027] The first liquid layer is a result of rapidly dissolving boron coated on the surface of the cathode active material in water, and thus the first liquid layer is subjected to acid treatment to induce dissolution of remaining boron. In addition, through the acid treatment, acid concentration and solution viscosity can be maintained as same level as in the single extraction of boron.

[0028] The acid-treated solution of the first liquid layer may be analyzed by inductively coupled plasma optical emission spectroscopy (ICP-OES) to measure the boron concentration. The ICP-OES may be performed in a conventional manner in the art, for example, under the condition as exemplified in the following Example. The content (A) of boron coated on the surface of the cathode active material may be calculated using the boron concentration as measured above.

[0029] Then, to extract boron present at grain boundary of the cathode active material sample, the first precipitate is dissolved in water to obtain a second liquid layer and a second insoluble precipitate, and then the second liquid layer is treated with acid (S3). At this time, dissolving the first precipitate in water may be performed at room temperature for 1 to 10 hours.

[0030] The acid-treated solution of the second liquid layer may be analyzed by ICP-OES to measure the boron concentration, and the content (B) of boron present at grain boundary of the cathode active material may be calculated using the boron concentration as measured above.

[0031] Then, to extract boron doped in lattice of the cathode active material sample, the second precipitate is dissolved by adding acid and hydrogen peroxide (S4). At this time, dissolving the second precipitate may be performed for 1 to 5 hours.

[0032] The solution obtained by treating the second precipitate with acid and hydrogen peroxide is analyzed by ICP-OES to measure the boron concentration, and the content (C) of boron doped in lattice of the cathode active material may be calculated using the boron concentration as measured above.

[0033] The content (C) of boron doped in lattice of the active material particles which is determined through extraction and ICP-OES analysis in a sequential process corresponds to a value according to Equation 1 below:

$$\text{Boron content in lattice (C)} = \text{Total boron content in the sample (D)} - (\text{Boron content in the coating layer (A)} + \text{Boron content at grain boundary (B)}) \quad [\text{Equation 1}]$$

[0034] In the steps of extracting boron for each distribution position, the acid that may be used for the first liquid layer, the second liquid layer and the second precipitate may be hydrochloric acid, and the acid may be used in an amount of 0.01 to 10 ml, preferably 5 to 10 ml based on 1 g of each object to be treated.

[0035] Meanwhile, the hydrogen peroxide is used to extract remaining boron that has not been dissolved by the acid, and the acid and hydrogen peroxide may be used in a volume ratio of 1:0.3 to 1:0.7, for example, 1:0.5.

[0036] As described above, when boron introduced into the cathode active material is sequentially extracted and analyzed by using a difference in solubility in water and an acid, it is possible to quantify the amount of boron present in each position according to the distribution pattern of boron in the cathode active material, thereby identifying the correlation between the performance improvement and the amount of boron introduced. From this, it can be confirmed that the optimal content of boron that contributes to the performance improvement of the cathode material.

Mode for Carrying out the Invention

[0037] Hereinafter, examples will be described in detail for helping understanding of the present invention.

Example 1: Extraction and content analysis according to distribution position of boron introduced into cathode active material

(Step 1) Preparation of cathode active material sample

[0038] $\text{LiNi}_{0.78}\text{Mn}_{0.11}\text{Co}_{0.11}\text{O}_2$ as a cathode active material and H_3BO_3 as a boron-containing compound were mixed in a dry mixer (CYCLOMIX, HOSOKAWA Micron Corporation), and then the resulting powders were fired at 150 °C for

5 hours. Through this process, three samples with boron introduced into the cathode active material were obtained, wherein the cathode active material and the boron-containing compound are applied to the sample in an amount as shown in Table 1, respectively.

(Step 2) Extraction and content measurement of boron present in coating layer of cathode active material

[0039] 0.1 g of each of the three samples obtained in step 1 was aliquoted and put into a vial, and it was dissolved by adding 20 g of ultrapure water thereto and then shaking at room temperature for 5 minutes. The resulting solution was allowed to stand so that a first liquid layer and a first precipitate (insoluble) were separated, and then filtered through a 0.45 μm filter.

[0040] After filtration, 0.5 ml of concentrated hydrochloric acid and 0.1 ml of an internal standard solution (Sc 1000 $\mu\text{g/ml}$) were added to 10 g of the first liquid layer, and the resulting solution was analyzed by ICP-OES. At this time, the ICP-OES (AVIO500, Perkin Elmer) was operated with the following conditions: Forward Power 1300 W; Torch Height 15 mm; Plasma gas flow rate 15.00 L/min; Sample gas flow rate 0.8 L/min; Auxiliary gas flow rate 0.20 L/min and Pump speed 1.5 ml/m.

[0041] From the concentration of boron measured by the ICP-OES, the content (A) of boron present in the coating layer of the cathode active material was calculated.

(Step 3) Extraction and content measurement of boron present at grain boundary of cathode active material

[0042] 0.1 g of the first precipitates (insoluble) separated in step 2 were put into a vial and dissolved by adding 20 g of ultrapure water thereto and then shaking at room temperature for 5 hours. The resulting solution was allowed to stand so that a second liquid layer and a second precipitate (insoluble) were separated, and then filtered through a 0.45 μm filter.

[0043] After filtration, 0.5 ml of concentrated hydrochloric acid and 0.1 ml of an internal standard solution (Sc 1000 $\mu\text{g/ml}$) were added to 10 g of the second liquid layer, and the resulting solution was analyzed by ICP-OES under the same conditions as in step 2.

[0044] From the concentration of boron measured by the ICP-OES, the content (B) of boron present at grain boundary of the cathode active material was calculated.

(Step 4) Extraction and content measurement of boron in lattice of cathode active material

[0045] 0.1 g of the second precipitates (insoluble) separated in step 3 were put into a vial and dissolved by adding 1 ml of concentrated hydrochloric acid and 0.5 ml of hydrogen peroxide at room temperature for 3 hours, and bubbles and heat were generated during the dissolution process.

[0046] 0.1 ml of an internal standard solution (Sc 1000 $\mu\text{g/ml}$) was added to the resulting solution, and ICP-OES analysis was performed under the same conditions as in step 2.

[0047] From the concentration of boron measured by the ICP-OES, the content (C) of boron in lattice of the cathode active material was calculated.

Comparative Example 1: Single extraction and content analysis of boron introduced into cathode active material

[0048] 0.1 g of each of the three samples obtained in step 1 of Example was aliquoted and put into a vial, and it was dissolved by adding 1 ml of concentrated hydrochloric acid and 0.5 ml of hydrogen peroxide at room temperature for 3 hours, and bubbles and heat were generated during the dissolution process.

[0049] 0.1 ml of an internal standard solution (Sc 1000 $\mu\text{g/ml}$) was added to the resulting solution, and ICP-OES analysis was performed under the same conditions as in step 2.

[0050] From the concentration of boron measured by the ICP-OES, the content (D) of boron introduced into the cathode active material was calculated.

[0051] The analysis results according to Example and Comparative Example are shown in Table 1 below.

[Table 1]

	Boron content (mg/kg of cathode active material)	Sample 1	Sample 2	Sample 3
	Amount used in sample preparation	1000	1000	1000
Example 1	Coating boron (A)	1035	420	540
	Boundary boron (B)	<10	<10	<10
	Lattice boron (C)	<10	45	125
Comparative Example 1	Total boron (D)	1035	465	665

[0052] From Table 1, even if the amount of boron used in preparing the sample was the same, the content of the coated or doped boron in each sample was different depending on reactivity between boron and the active material (especially, for Sample 1, it was found that the content in the active material was different from the amount of boron introduced due to the weight error when weighing boron raw materials or the purity of boron). By sequentially extracting boron using the difference in solubility in water and an acid according to the distribution position in the active material, the content of boron to penetrate to the inside could be measured. That is, it can be seen that, in Sample 1, most of the boron introduced into the cathode active material is present in the coating layer (A) on the surface, and in Samples 2 and 3, the introduced boron is dispersed and distributed in the coating layer (A), at grain boundary (B) and in lattice (C). Through the distribution pattern of boron, it is possible to determine the optimal content of boron required to improve the performance of the cathode active material.

[0053] In addition, from the results of Comparative Example, it was confirmed that the content (C) of boron in lattice of the cathode active material particles corresponds to a value according to Equation 1 below.

$$\text{Boron content in lattice (C)} = \text{Total boron content in the sample (D)} - (\text{Boron content in the coating layer (A)} + \text{Boron content at grain boundary (B)}) \quad [\text{Equation 1}]$$

[0054] Therefore, as in Example 1, when boron introduced into the cathode active material is extracted and analyzed according to the distribution position, it is possible to quantify the amount of boron present in each position according to the distribution pattern of boron in the cathode active material, thereby identifying correlation between the performance improvement and the amount of boron introduced. From this, it can be determined the optimal content of boron that contributes to the performance improvement of the cathode material.

Example 2:

[0055] In order to determine extraction content of boron and lithium according to the dissolution time in water of the cathode active material into which boron is introduced, $\text{LiNi}_{0.78}\text{Mn}_{0.11}\text{Co}_{0.11}\text{O}_2$ and H_3BO_3 were dry mixed and fired as in Step 1 of Example 1 to produce a sample (1000 mg of boron/1kg of cathode active material), and then 0.1 g of the sample was aliquoted and put into a vial and it was dissolved by adding 20 g of ultrapure water thereto at room temperature, and ICP-OES analysis was performed under the same conditions as in Example 1 depending on the dissolution time.

[0056] As a result, the contents of boron and lithium depending on the dissolution time are shown in Table 2 and Fig. 2.

[Table 2]

Dissolution time (hr)	Content (mg/1 kg of cathode active material)		Li dissolution rate (corresponds to the straight line slope of Fig. 2)
	B	Li	
0.1	1068	1470	
1	1084	1786	314
5	1093	2076	72
22	1093	2511	25.6
45	1105	3052	23.5

[0057] From Table 2 and Fig. 2, the content (extraction concentration) of boron is not significantly changed according to the dissolution time and most of boron is present in the coating, while the slope of the content (extraction concentration)

of lithium is divided into three stages according to the dissolution time and the lithium results are similar to the results for Samples 2 and 3 used in Example 1. These results will be used as indirect evidence that the lithium applied to the cathode active material at a high concentration has different distribution pattern in the active material particles depending on the dissolution rate. The difference in the dissolution rate of the lithium component is presumed to be due to partial desorption and dissolution of lithium from the structure of the NCM-based cathode active material (Li[Ni/Co/Mn]O₂).

Claims

1. An analysis method of content of boron introduced into a cathode active material, the method comprising:

(S1) preparing a cathode active material sample into which boron is introduced;
 (S2) dissolving the cathode active material sample in water to obtain a first liquid layer and a first precipitate and treating the first liquid layer with acid, and then analyzing the resulting solution by inductively coupled plasma optical emission spectroscopy (ICP-OES) to determine the concentration of boron;
 (S3) dissolving the first precipitate in water to obtain a second liquid layer and a second precipitate and treating the second liquid layer with acid, and then analyzing the resulting solution by ICP-OES to determine the concentration of boron; and
 (S4) adding acid and hydrogen peroxide to the second precipitate, and then analyzing the resulting solution by ICP-OES to determine the concentration of boron.

2. The analysis method according to claim 1, wherein the cathode active material sample is prepared by dry mixing a boron-containing compound and a cathode active material, followed by firing, such that boron is present on the surface, at grain boundary, and in lattice of the cathode active material particles.

3. The analysis method according to claim 2, wherein the boron-containing compound comprises at least one compound selected from the group consisting of H₃BO₃, B₂O₃, C₆H₅B(OH)₂, (C₆H₅O)₃B, [CH₃(CH₂)₃O]₃B, C₁₃H₁₉BO₃, C₃H₉B₃O₆, and (C₃H₇O)₃B.

4. The analysis method according to claim 2, wherein the cathode active material comprises a lithium transition metal oxide represented by the following formula 1:



wherein M is one or two or more selected from the group consisting of Al, Zr, Zn, Ti, Mg, Ga and In; $0 \leq x \leq 1.0$, $0 \leq y \leq 0.6$, $0 \leq z \leq 0.6$ and $0 \leq v \leq 0.1$.

5. The analysis method according to claim 2, wherein the firing is carried out at a temperature of 130 °C to 300 °C.

6. The analysis method according to claim 1, wherein the amount of the acid used in the steps S2 to S4 is a range of 0.01 to 10 ml based on 1 g of the object to be treated.

7. The analysis method according to claim 1, wherein the acid used in the steps S2 to S4 comprises hydrochloric acid.

8. The analysis method according to claim 1 or 2, wherein the method comprising:

calculating a content (A) of boron coated on the surface of the cathode active material using the boron concentration measured in step S2,
 calculating a content (B) of boron present at grain boundary of the cathode active material using the boron concentration measured in step S3, and
 calculating a content (C) of boron doped in lattice of the cathode active material using the boron concentration measured in step S4.

9. The analysis method according to claim 8, wherein the content (C) of boron present in lattice of the cathode active material corresponds to a value according to Equation 1 below:

Boron content in lattice (C) = Total boron content in the sample (D) - (Boron content in the coating layer (A) + Boron content at grain boundary (B)) [Equation 1]

Patentansprüche

1. Verfahren zur Analyse des Gehalts von Bor, das in ein Kathodenaktivmaterial eingebracht ist, wobei das Verfahren umfasst:

(S1) Herstellen einer Kathodenaktivmaterialprobe, in die Bor eingebracht ist,
 (S2) Lösen der Kathodenaktivmaterialprobe in Wasser, um eine erste Flüssigkeitsschicht und einen ersten Niederschlag zu erhalten, und Behandeln der ersten Flüssigkeitsschicht mit Säure und dann Analysieren der resultierenden Lösung durch optische Emissionsspektroskopie mit induktiv gekoppeltem Plasma (ICP-OES), um die Borkonzentration zu bestimmen,
 (S3) Lösen des ersten Niederschlags in Wasser, um eine zweite Flüssigkeitsschicht und einen zweiten Niederschlag zu erhalten, Behandeln der zweiten Flüssigkeitsschicht mit Säure und dann Analysieren der resultierenden Lösung durch ICP-OES, um die Borkonzentration zu bestimmen, und
 (S4) Zugeben von Säure und Wasserstoffperoxid zum zweiten Niederschlag und dann Analysieren der resultierenden Lösung durch ICP-OES, um die Borkonzentration zu bestimmen.

2. Analyseverfahren gemäß Anspruch 1, wobei die Kathodenaktivmaterialprobe durch Mischen einer borhaltigen Verbindung und eines Kathodenaktivmaterials, gefolgt von Brennen, so hergestellt wird, dass Bor an der Oberfläche, an Korngrenzen und im Gitter Kathodenaktivmaterialpartikel vorhanden ist.

3. Analyseverfahren gemäß Anspruch 2, wobei die borhaltige Verbindung mindestens eine Verbindung, ausgewählt aus der Gruppe, bestehend aus H_3BO_3 , B_2O_3 , $C_6H_5B(OH)_2$, $(C_6H_5O)_3B$, $[CH_3(CH_2)_3O]_3B$, $C_{13}H_{19}BO_3$, $C_6H_9B_3O_6$ und $(C_3H_7O)_3B$, umfasst.

4. Analyseverfahren gemäß Anspruch 2, wobei das Kathodenaktivmaterial ein durch die folgende Formel 1 dargestelltes Lithiumübergangsmetalloxid umfasst:



worin M eines oder zwei oder mehr, ausgewählt aus der Gruppe, bestehend aus Al, Zr, Zn, Ti, Mg, Ga und In, ist, $0 \leq x \leq 1,0$, $0 \leq y \leq 0,6$, $0 \leq z \leq 0,6$ und $0 \leq v \leq 0,1$ ist.

5. Analyseverfahren gemäß Anspruch 2, wobei das Brennen bei einer Temperatur von 130°C bis 300°C durchgeführt wird.

6. Analyseverfahren gemäß Anspruch 1, wobei die Menge der in den Schritten S2 bis S4 verwendeten Säure ein Bereich von 0,01 bis 10 ml, bezogen auf 1 g des zu behandelnden Objekts, ist.

7. Analyseverfahren gemäß Anspruch 1, wobei die in den Schritten S2 bis S4 verwendete Säure Salzsäure umfasst.

8. Analyseverfahren gemäß Anspruch 1 oder 2, wobei das Verfahren umfasst:

Berechnen eines Gehalts (A) an Bor, das auf der Oberfläche des Kathodenaktivmaterials beschichtet ist, unter Verwendung der in Schritt S2 gemessenen Borkonzentration,
 Berechnen eines Gehalts (B) an Bor, das an Korngrenzen des Kathodenaktivmaterials vorhanden ist, unter Verwendung der in Schritt S3 gemessenen Borkonzentration und
 Berechnen eines Gehalts (C) an Bor, das im Gitter des Kathodenaktivmaterials dotiert ist, unter Verwendung der in Schritt S4 gemessenen Borkonzentration.

9. Analyseverfahren gemäß Anspruch 8, wobei der Gehalt (C) an Bor, das im Gitter des Kathodenaktivmaterials vorhanden ist, einen Wert gemäß der nachstehenden Gleichung 1 entspricht:

Borgehalt im Gitter (C) = Gesamtborgehalt in der Probe (D) - (Borgehalt in der Beschichtungsschicht (A) + Borgehalt an Korngrenzen (B)).

[Gleichung 1]

Revendications

1. Procédé d'analyse de la teneur en bore introduit dans un matériau actif de cathode, le procédé comprenant :

(S1) préparer un échantillon de matériau actif de cathode dans lequel le bore est introduit ;
 (S2) dissoudre l'échantillon de matériau actif de cathode dans de l'eau afin d'obtenir une première couche liquide et un premier précipité et traiter la première couche liquide avec de l'acide, puis analyser la solution résultante par spectroscopie d'émission optique à plasma à couplage inductif (ICP-OES) afin de déterminer la concentration en bore ;
 (S3) dissoudre le premier précipité dans de l'eau afin d'obtenir une seconde couche liquide et un second précipité et traiter la seconde couche liquide avec de l'acide, puis analyser la solution résultante par ICP-OES afin de déterminer la concentration en bore ; et
 (S4) ajouter de l'acide et du peroxyde d'hydrogène au second précipité, puis analyser la solution résultante par ICP-OES afin de déterminer la concentration en bore.

2. Procédé d'analyse selon la revendication 1, dans lequel l'échantillon de matériau actif de cathode est préparé par mélange à sec d'un composé contenant du bore et d'un matériau actif de cathode, suivi d'une cuisson, de telle sorte que le bore soit présent sur la surface, au niveau du joint de grains, et dans le réseau des particules de matériau actif de cathode.

3. Procédé d'analyse selon la revendication 2, dans lequel le composé contenant du bore comprend au moins un composé choisi dans le groupe constitué de H_3BO_3 , B_2O_3 , $C_6H_5B(OH)_2$, $(C_6H_5O)_3B$, $[CH_3(CH_2)_3O]_3B$, $C_{13}H_{19}BO_3$, $C_3H_9B_3O_6$ et $(C_3H_7O)_3B$.

4. Procédé d'analyse selon la revendication 2, dans lequel le matériau actif de cathode comprend un oxyde de métal de transition au lithium représenté par la formule 1 suivante :



dans lequel

M est un ou deux éléments ou plus choisis dans le groupe constitué de Al, Zr, Zn, Ti, Mg, Ga et In ; $0 \leq x \leq 1,0$, $0 \leq y \leq 0,6$, $0 \leq z \leq 0,6$ et $0 \leq v \leq 1$.

5. Procédé d'analyse selon la revendication 2, dans lequel la cuisson est réalisée à une température de 130 °C à 300 °C.

6. Procédé d'analyse selon la revendication 1, dans lequel la quantité d'acide utilisé dans les étapes S2 à S4 est dans une plage de 0,01 à 10 ml sur la base de 1 g de l'objet à traiter.

7. Procédé d'analyse selon la revendication 1, dans lequel l'acide utilisé dans les étapes S2 à S4 comprend de l'acide chlorhydrique.

8. Procédé d'analyse selon la revendication 1 ou la revendication 2, dans lequel le procédé comprend :

calculer une teneur (A) en bore déposé sur la surface du matériau actif de cathode en utilisant la concentration en bore mesurée dans l'étape S2,
 calculer une teneur (B) en bore présent au niveau du joint de grains du matériau actif de cathode en utilisant la concentration en bore mesurée dans l'étape S3, et
 calculer une teneur (C) en bore dopé dans le réseau du matériau actif de cathode en utilisant la concentration en bore mesurée dans l'étape S4.

9. Procédé d'analyse selon la revendication 8, dans lequel la teneur (C) en bore présent dans le réseau du matériau

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actif de cathode correspond à une valeur conformément à l'équation 1 ci-dessous :

$$5 \quad \text{Teneur en bore dans le réseau (C) = Teneur totale en bore dans l'échantillon (D) - (Teneur en bore dans la couche de dépôt (A) + Teneur en bore au niveau du joint de grains (B)).} \quad [\text{Equation 1}]$$

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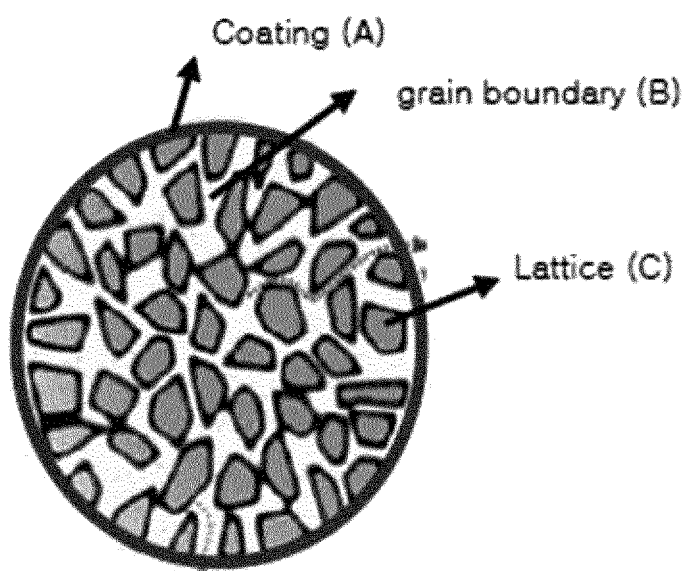
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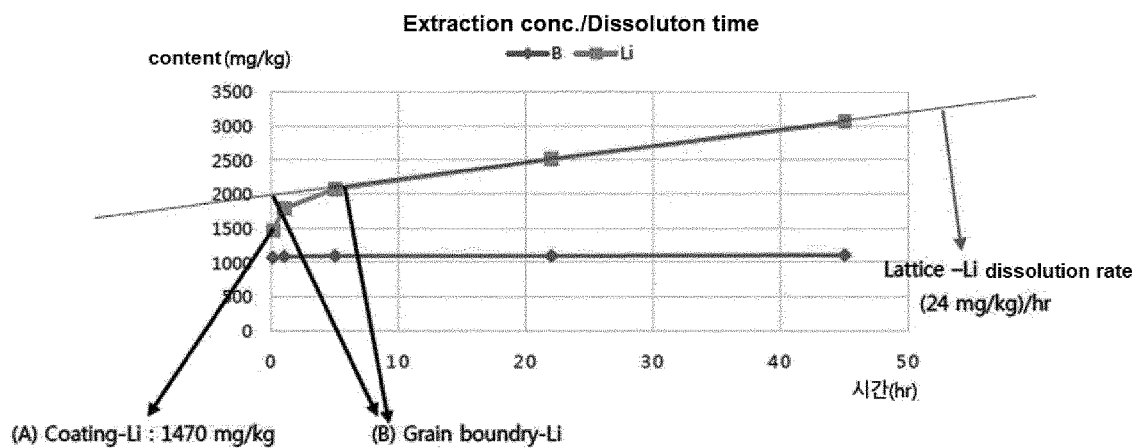
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[Fig. 1]



[Fig. 2]



REFERENCES CITED IN THE DESCRIPTION

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